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Microstructural and Magnetic Observations of Compacted FeCoV Nanoparticles

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Abstract—One attempt to improve core loss of electromagnetic machines is to utilize nanocrystalline alloys, which are predicted to have extremely soft magnetic properties. High magnetic saturation values and high Curie temperatures make iron-cobalt alloys attractive for such applications. In this work iron-cobalt-vanadium nanopowders were synthesized and compacted. The coated particles were characterized and examined in both the powder and compacted states. Higher than expected coercivities are reported and may be due to the inhomogeneous microstructure resulting from interactions with the vanadium.

Index Terms—Characterizations, iron-cobalt alloys, microstructure, nanopowders.

I. INTRODUCTION

EXTREMELY soft magnetic properties of partially crystallized amorphous precursors are well understood and can be explained by Herzer's Modified Random Anisotropy Model [1]. It is also possible to produce nanocrystalline structures by beginning with powder precursors followed by a densification process. This idea was employed in the development of nanocrystalline soft bulk magnets for high temperature power applications. FeCo alloys are attractive due to their higher induction and magnetic-nonmagnetic transition temperatures. For a nanocrystalline structure to be able to operate at elevated temperatures, grain growth must be minimized. It was found that a secondary stable phase at the grain boundaries is necessary to control the grain growth. The electromechanical machine designer may take advantage of this grain boundary requirement by introducing a highly resistive secondary phase, such as an oxide or carbide, to further reduce eddy current losses associated with alternating fields. However, this resistive coating should be thin enough to still allow magnetic coupling of neighboring grains.

II. EXPERIMENTAL PROCEDURES

A detailed description of the thermal plasma synthesis of oxide-coated and carbon-coated FeCo nanocrystalline powders has been previously reported [2]. The alloy investigated under this effort, however, contained additional constituents to include 1.8% vanadium. A controlled amount of air was introduced

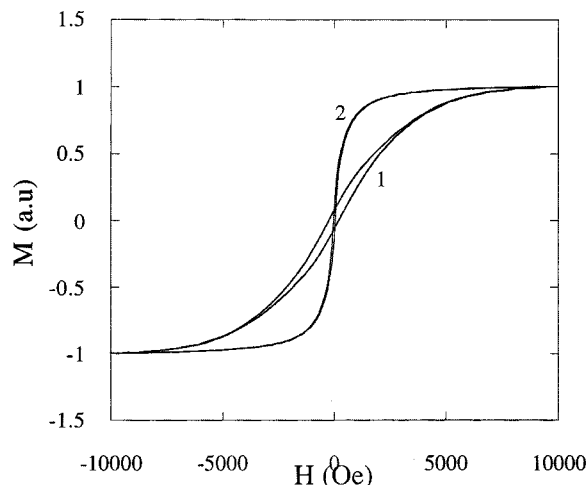


Fig. 1. As produced (1) and compacted (2) hysteresis loops.

into the plasma stream to produce a thin protective oxide layer. Conventional x-ray diffractions ($\text{Cu } K_{\alpha}$) as well as a Synchrotron x-ray analysis were used to search for the presence of an oxide layer. Magnetic saturation, M_s , and coercivities, H_c , were measured using a Lake Shore 7300 vibrating sample magnetometer. As-produced powders were evacuated and sealed in Pyrex ampoules at 675°C for 45 minutes to remove any gas that may have been trapped during synthesis. They were then Hot Isostatically Pressed (HIP'ed) at 725°C for 1 h at a pressure of 151 MPa. Microscopy studies were performed with a Philips FEG-30 Scanning Electron Microscope (SEM) and a JEOL 120-CX Transmission Electron Microscope (TEM).

III. RESULTS AND DISCUSSION

X-ray diffraction, using K_{α} radiation from a copper source, on as produced powders did not indicate the presence of an oxide. However, the presence of a CoFe_2O_4 oxide was detected by Synchrotron X-ray analysis with energies tuned to the absorption edges of Fe and Co, which are very close to each other. Nanopowders synthesized with different plasma settings yielded magnetic saturation values (M_s) between 185–215 emu/g and coercivities (H_c) at around 290 Oe, at room temperature.

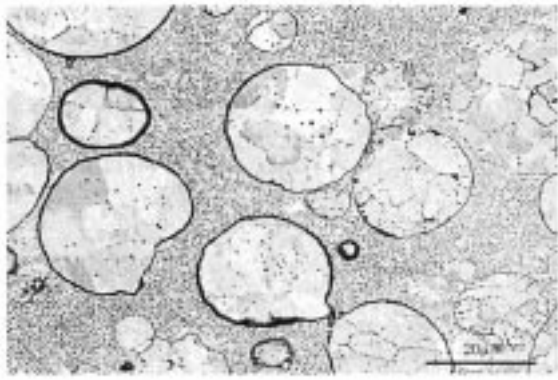
A detailed microstructural analysis on the compacted nanoparticles was necessary to develop a better understanding of the correlation between magnetic properties and microstructure. After compaction, the higher coercivities observed in powder form and reported previously [2] are reduced to 39 Oe, as illustrated in Fig. 1, which is still many times larger than the coercivity for bulk FeCo alloys. The depicted hysteresis loops

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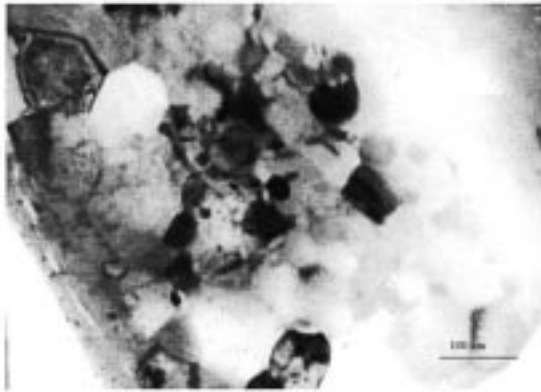
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(a)



(b)

Fig. 2. SEM image (a) and TEM image (b) of compacted oxide-coated FeCo nanoparticles.

indicate a difference in the reversal mechanism associated with the powder and compacted forms. While in powder form, the rotation process dominates in the absence of domain walls. In bulk form the reversal mechanism is due to the domain wall motion.

Microstructural analysis performed on the SEM indicated that compaction yielded 100% dense structures (Fig. 2(a))

with a segregation of vanadium giving rise to circular grain like structures. Thermomagnetic measurements on compacted samples showed no presence of a ferrite phase indicating that the vanadium reduces all of the ferrite. Systems studied earlier, without V, showed a small bump in $M(T)$ around 520°C, which is the Néel temperature of the ferrite phase. Energy Dispersive X-ray (EDX) analysis indicated that the darker regions are vanadium and oxygen rich while lighter regions are free of those elements. Therefore, it is speculated that vanadium diffuses through the surface and reduces the ferrite to form vanadium-oxide. SEM analysis on pre-annealed powders confirmed that these larger features form during the degassing process prior to compaction. The areas surrounding these larger figures have a uniform distribution of the vanadium-oxide phase. TEM studies of compacted FeCo samples containing no Vanadium indicated that the structure is in fact still nanocrystalline as can be seen in Fig. 2(b).

IV. SUMMARY

While a simple model developed by Néel [3] can be used to explain the origin of higher coercivities in powder form, the compacted form does not seem to follow Herzer's Modified Random Anisotropy Model for partially crystallized amorphous precursors. It may be this inhomogeneity in microstructure that is responsible for higher coercivity values and it is believed that in regions where vanadium is disclosed, grain growth kinetics are faster making the material magnetically harder. On the other hand, in regions where the secondary vanadium phase is located on the grain boundaries, a crystalline interface may induce an interface anisotropy as opposed to having an amorphous interface in partially crystallized amorphous precursors. In this case one may expect different behavior than predicted by Herzer.

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